Bulgarian Chemical Communications, Volume 52, Issue 3 (pp. 369-373) 2020 *DOI: 10.34049/bcc.52.3.5236*

Research of tribological parameters of multilayer coating Ti/TiN/CrN-ml deposited on 1.2343 steel

V. Rupetsov[1*], S. Dishliev[2], G. Mishev[1], F. Franek[3], M. Premauer[3], L. Kolakleva[4]

[1] *Plovdiv University "Paisii Hilendarski", 24 Tzar Assen Str., 4000 Plovdiv, Bulgaria,*
[2] *University of Food Technologies, 26 Maritsa Blvd. 4000 Plovdiv, Bulgaria*
[3] *Austrian Excellence Center for Tribology, Viktor-Kaplan-Str. 2/C, 2700 Wiener Neustadt, Austria*
[4] *Central Laboratory of Applied Physics, Bulgarian Academy of Sciences, 61 Sankt Petersburg Blvd. 4000 Plovdiv, Bulgaria*

Received: February 25, 2020; Accepted: May 11, 2020

This work is dedicated to the experimental investigation of the tribological parameters of multilayer coating Ti/TiN/CrN-ml deposited on 1.2343 (X37CrMoV5-1) steel. This steel is mainly used for injection molds production. The improval of their wear resistance increases their living resources and betters the economic parameters of the company. Multilayer coating Ti/TiN/CrN-ml was deposited by the PVD method - reactive magnetron sputtering in Nanotech Group Ltd. - Plovdiv. It has a nanolaminate structure with a symmetric bylayer period of 22-30 µm. Methodology for experimental investigation of the tribological parameters of the multilayer coating Ti/TiN/CrN-ml is proposed which is based on "Ball on Flat Sliding Wear Test" tribosystem and determines the volume of the wear trace. The dimensions of the trace were determined by trace topography also using a microscope TESA VISIO-300. Coating thickness, nanohardness, friction coefficient and modulus of elasticity were measured. The influence of the main factors of the tribosystem (normal load, path and sliding speed) on the wear intensity is described.

Keywords: multilayer coating, wear intensity, PVD method

INTRODUCTION

Injection molds work under very hard regimes: high loads; high temperatures; high wear [1-4]. The tool resource is directly related to the wear of working surfaces. In order to increase the resource (life) of the injection molds, which is going to lead to a significant economic effect, it is necessary to take measures for increasing the hardness of the working surfaces of the tool and improving the wear resistance thereof [5]. About the enhancement of these two properties, the methods for deposition of hard wear-resistant coatings are the most effective [6-11]. Recently, the most widely use possess the coatings obtained by the process of Physical Vapor Deposition (PVD) [12, 13].

EXPERIMENTAL

Samples with cuboid shape: 25 × 8 × 5 mm were prepared as follows (used face: 25 × 8 mm):

- Unhardened ground (marked as 2343A) with hardness 145 HB and surface roughness Ra 0.165 µm;
- Hardened ground (marked as 2343B) with hardness 52 HRC and surface roughness Ra 0.112 µm;
- Hardened polished (marked as 2343C) with hardness 52 HRC and surface roughness Ra 0.033 µm.

Static friction coefficient of the coating is 0.25±0.03 (measured against polished SS 304 L). The common thickness of the layer is 3.6±0.05 µm. First, it had a 0.9 µm adhesive layer consisting of Ti, followed by gradient TiN and TiCrN nanolayers. After that a nanolaminate was composed by alternating layers of TiN and CrN with thickness of 11÷15 nm each. The last layer is gradient 0.5 µm thick, which is changed from CrN through TiCrN to TiN (150 nm thick). The nanolaminate structure has the greatest influence on the coating properties and thus the signature "Ti/TiN/CrN-ml" was used (with the consent of the manufacturing company [14]). The temperature during the deposition process was 170°C.

Methods for experimental investigation of the tribological characteristics of the test samples

- *Method for experimental investigation of the wear resistance of thin hard coatings.*

The experimental studies were conducted by the "Ball on Flat Sliding Wear Test" friction scheme in horizontal orientation of the test surface [15]. One cemented-oxide ball (Al_2O_3) with diameter of 3 mm, fixed in a holder, was used as a counter-part. It rubs a linear reciprocating sample without lubricant, working in air at room temperature. The width of the arisen grooves was measured by a microscope: PC based contactless measurement

---

* To whom all correspondence should be sent:
E-mail: velko_r@abv.bg

system TESA VISIO-300 at 100 × magnification (resolution: 0.001 mm).

The average width value b_{cp} was calculated:

$$b_{cp} = \frac{1}{n}\sum_{i=1}^{n} b_i \text{ , mm} \qquad (1)$$

where:

n – number of the grooves (traces);

b_i – width of every particular groove.

For control of the measured width of the traces the topography of the sample surface in the traces area was generated (Figure 1). In accordance with the topographic results it was found that the error of measurement is less than 1%, which gives us to believe that the obtained results are correct.

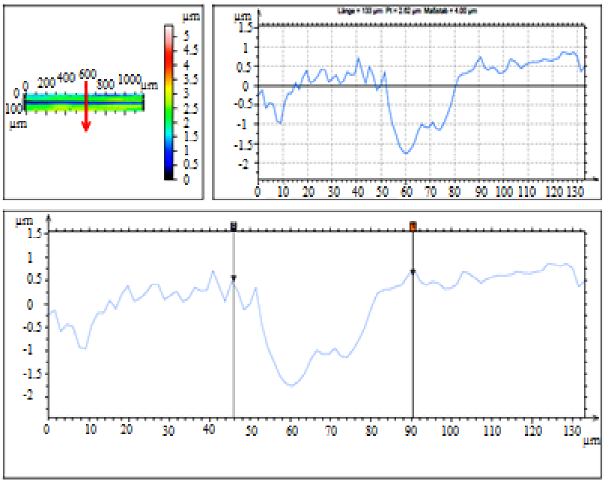

Fig. 1. Surface topography in the traces area

Traces width was used to determine the wear volume or volume of the traces which determines the wear rate I_w by the equation:

$$I_w = \frac{V}{F.L}\text{, } mm^3/N.m \qquad (2)$$

where:

V – wear volume (volume of the trace), mm^3;

F – normal load upon the ball, N;

L - sliding distance between the ball and the sample, m.

The volume of the track was determined by the methodology described in [16].

Methods for investigation of thickness, nanohardness, elastic modulus and adhesion of the coating

The coating thickness h is a key complex indicator and was measured by a calotester elaborated in CLAP-Plovdiv. A ball with a diameter of 30 mm was implemented and the value of h was calculated by the well-known formula [17,18]:

$$h = \frac{D^2 - d^2}{8R}.10^3 \text{ , µm} \qquad (3)$$

where:

D - outer section diameter, *mm*;

d - inner section diameter, *mm*;

R - ball radius, *mm* (here: 15 *mm*).

The nanohardness and elastic modulus of the coating were determined using Compact Platform CPX (MHT/NHT) CSM Instruments in CLAP-Plovdiv. A diamond indenter (Berkovich type) was used and the results were interpreted by the Oliver and Pharr method [19].

The adhesion was tested by a Micro Scratch Tester (MST) module included in the same apparatus. A diamond indenter (Rockwell type) with a rounded apex of 200 µm was used [20].

RESULTS AND DISCUSSION

Experimental study of the wear resistance of a multilayer Ti/TiN/TiCrN-ml on 1.2343 steel

- *Experimental results for the influence of thenormal load on the wear rate.*

Experimental studies were conducted under the following constant tribosystem parameters: sliding speed $V=10$ *mm/s*; path $L=50$ *m*. The normal load varied from *1* to *5 N*. The wear of the investigated coating depends mainly of the normal load.

The experimental results were processed using the correlation analysis method. The calculated coefficient of determination shows the strength of the mutual relationships between I_w and F. For each value of normal load, three trials were performed and the mean arresting values were taken. In Table 1 the correlation equations of the wear intensity as a function of the normal load for all types of samples (12_2343A; 12_2343B; 12_2343C) and the corresponding coefficient of determination are given.

Table 1. Correlation dependencies of wear rate as a function of normal load.

Sample	Correlation equation	Coefficient of determination
12_2343A	$I_w=4.390.F+4.374$	$R^2=0.999$
12_2343B	$I_w=2.753.F+4.999$	$R^2=0.874$
12_2343C	$I_w=2.276.F-2.033$	$R^2=0.770$

At a coefficient of determination $R^2>0.7$ the mutual relation between I_w and F is very strong.

Figure 2 shows the dependence of I_w on F. With normal load increasing, the wear intensity also increases.

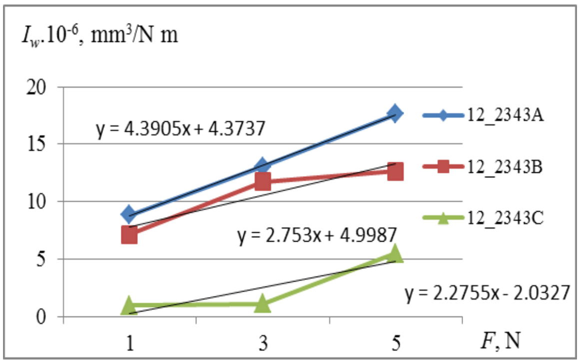

Fig. 2. Graphic dependencies of the wear rate as a function of the load $I_w=f(F)$

- *Experimental results for the influence of the path on the wear rate.*

Experimental studies were conducted under the following constant tribosystem parameters: sliding speed *V=10 mm/s*; normal load *F=1 N*. The path varied from *50* to *100 m*.

The experimental results were processed using the correlation analysis method.

In Table 2 the correlation equations of the wear intensity as a function of the path for all types of samples (12_2343A; 12_2343B; 12_2343C) and the corresponding coefficient of determination are given.

Table 2. Correlation dependencies of wear rate as a function of the path.

Sample	Correlation equation	Coefficient of determination
12_2343A	$I_w=3.068.L+6.043$	$R^2=0.976$
12_2343B	$I_w=-1.438.L+8.371$	$R^2=0.937$
12_2343C	$I_w=0.394.L+0.562$	$R^2=0.999$

The negative value in the correlation equation of sample 12_2343B indicates an inverse proportional dependence of I_w on L. When L increases, I_w decreases. Figure 3 shows the dependence of the wear rate as a function of the path.

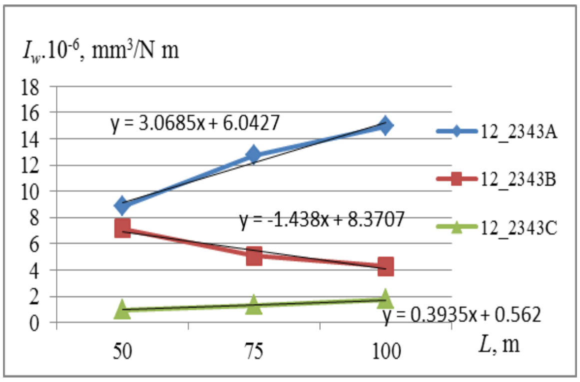

Fig. 3. Graphic dependencies of the wear rate as a function of the path $I_w=f(L)$.

- *Experimental results for the influence of the velocity on the wear rate.*

Experimental studies were conducted under the following constant tribosystem parameters: normal load *F=1N*; path *L=50 m*. The sliding speed varied from *5* to *15* mm/s. In Table 3 the correlation equations of the wear intensity as a function of the velocity for all types of samples (12_2343A; 12_2343B; 12_2343C) and the corresponding coefficient of determination are given.

Table 3. Correlation dependencies of wear rate as a function of the velocity.

Sample	Correlation equation	Coefficient of determination
12_2343A	$I_w=-0.852.V+11.513$	$R^2=0.506$
12_2343B	$I_w=-0.609.V+8.929$	$R^2=0.610$
12_2343C	$I_w=0.111.V+1.992$	$R^2=0.010$

Figure 4 shows the dependence of the wear rate as a function of the velocity.

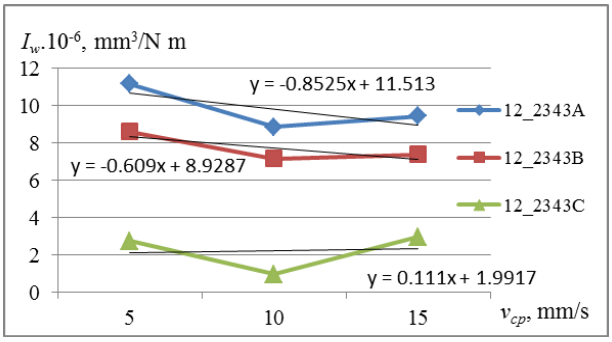

Fig. 4. Graphic dependencies of the wear rate as a function of the velocity $I_w=f(V)$.

Investigation of thickness, nanohardness, elastic modulus and adhesion of the coating

On the base of the calotester measurement, the calculated thickness is 2.92 µm. A nanoindentation curve (load L vs. indentation depth D) of the test sample 12_2343C is shown in Figure 5. These parameters were specified: nanohardness H = 25 GPa; elastic modulus E = 316 GPa; maximal penetration depth h_m = 336 nm (at maximal indentation load of 50 mN). The maximal penetration depth is less than 15% of the entire coating thickness (2.92 µm), which guarantees that the measured nanohardness is related only to coating without a substrate impact [21].

Figure 6a shows a diagram illustrating the adhesion measurement. The force F_n which presses the indenter is changed linearly to a maximum value of 30 N (the maximum load allowed by this equipment).

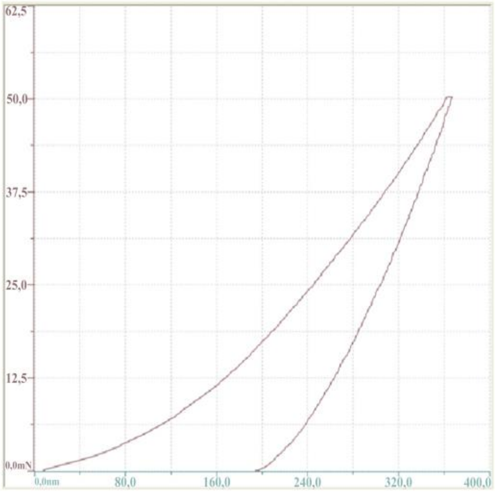

Fig. 5. Nanoindentation load/unload curve of sample 12_2343C [21]

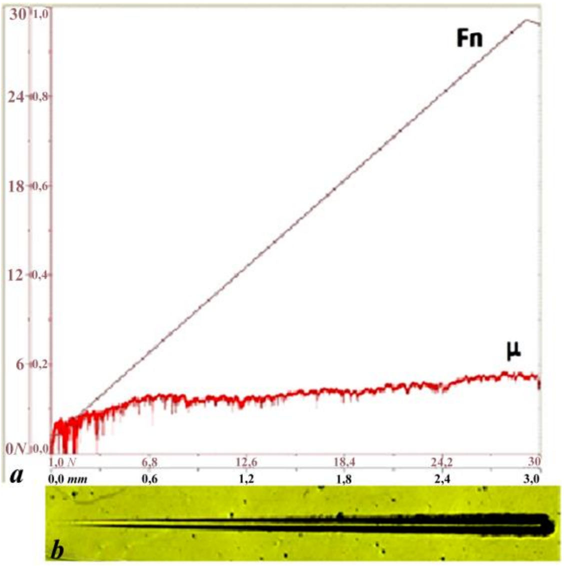

Fig. 6. Scratch test of sample 12_2343C: a) curves of the applied force F_n and friction coefficient μ; b) view of the trace [21].

Along the trace, the friction coefficient was measured. It can be seen that it is lower than the above-mentioned static friction coefficient (friction coefficient at rest). This is consistent with the theory in mechanics where the friction coefficient at rest always has a larger value. Also, there is a difference in friction bodies material in both cases (polished SS 304L and diamond). The friction coefficient value is substantially constant (in the region of the tripping to the first damage), which can be explained by small tensions in the coating because of its nanolaminate structure, since they are unloaded between the individual layers. The first obvious damages in the coating were originated by applied force of 24 N (Figure 6b). This is the first critical load Fc_1. Since a delamination of the coating (an exposure of the substrate) in the channel is not noticed, it is proven that the second critical force Fc_2 is not reached up to the maximal applied force (30 N) [21].

CONCLUSIONS

- The main factors of a tribosystem: normal load, path and velocity have a significant impact on the wear of the multilayer nanolaminat coating Ti/TiN/CrN-ml deposited on 1.2343 steel. With normal load and path increasing the coatings wear increases as well. With velocity increasing the coating wear decreases.

- The quality of the surfaces, on which Ti/TiN/CrN-ml coating is deposited, has a significant impact on the wear of the coating. This multilayer coating doesn’t change the initial surface roughness. When surface roughness decreases and/or hardness increases, the wear rate of the coating is reduced.

- The calculated wear rate of the Ti/TiN/CrN-ml coating deposited on a hardened polished surface is on average 4 times lower than that on a hardened ground surface and on average 6 times lower than that on an unhardened ground surface.

- The measured hardness and adhesion show a coating with a middle range hardness (on the edge of conventional limit for hard coatings: 20 GPa) and an excellent adhesion. Both parameters are benefited by this nanolaminate coating structure.

***Acknowledgements**: The authors would like to acknowledge the support of the National Programme "Young Scientists and Post-Doctoral Students" funded by the Bulgarian Ministry of Education and Science (MES).*

REFERENCES

1. H. Rees, Mold Engineering, 2nd edn., Munich, Hanser Publishers, 2002.
2. T. Osswald, L.-S. Turng, P. Gramann, Injection Molding Handbook, 2nd edn., Munich, Hanser Publishers, 2008.
3. P. Unger, Gastrow Injection Molds – 130 Proven Designs, 4th edn., Munich, Hanser Publishers, 2006.
4. W. C. Oliver, G. M. Pharr, *J. Mater. Res.*, **7** (6) 1564 (1992).
5. E. A. Levashov, Ensuring the unity of measurements of physicomechanical and tribological properties of nanostructured surfaces, MISiSISMAN, Moscow, 2010 (in Russian).
6. M. I. Jones, I. R. McColl, D. M. Grant, *Surf. Coat. Technol.*, **132** (2-3), 143 (2000).
7. M. J. Jung, K. H. Nam, L. R. Shaginyan, J. G. Han, *Thin Solid Films*, **435** (1-2), 145 (2003).

8. D. Kakaš, B. Škorić, A. Miletić, P. Terek, M. Vilotić, L. Kovačević, Proc. 7th Int. Conf. on Trib. (BALKANTRIB'11),Thessaloniki, Greece, 2011, p. 49.
9. P. Sveshtarov, R. Kakanakov, L. Kolaklieva, Ch. Bachchedjiev, T. Cholakova, E. Trifonova, S. Evtimova, Intern. Workshop NANOHARD, Velingrad, Bulgaria, 2007, p. 49.
10. S. Veprek, M. G. J. Veprek-Heijman P. Karvankova, J. Prochazka, *Thin Solid Films*, **476** (1), 1 (2004).
11. S. Zhang, D. Sun, Y. Fu, H. Du, *Surf. Coat. Technol.*, **167** (2-3) 113 (2003).
12. Ch. Pashinski, M. Angelov, V. Rupetsov, D. Petrov, P. Shindov, S. Dishliev, Proc. IV Int. Conf. Industr. Eng. Env. Protect. 2014 (IIZS 2014) Zrenjanin, Serbia, 2014, p. 356.
13. Ch. Pashinski, *Int. J. Sci. & Eng. Res.*, **4** (6), 870 (2013).
14. Nanotech Group Ltd., Coatings for molding and forming tools, available on www.nanotech-group.com/mouldingformingtools.html at 05. 08. 2019.
15. http://www.astm.org/Standards/G133.htm, ASTM G133 – 05 Standard Test Method for Linearly Reciprocating Ball-on-Flat Sliding Wear (2010).
16. V. S. Rupetsov, *Journal of Food and Packaging Science, Technique and Technologies*, **1**(4), 60 (2014) ISSN 1314-7773.
17. www.nanovea.com.
18. www.platit.com.
19. www.nanoscan.info.
20. www.csm-instruments.com.
21. V. S. Rupetsov, L. P. Kolaklieva, V. I Kopanov, V. A. Chitanov, Ch. O. Pashinski, S. I. Dishliev, *Journal of Food and Packaging Science, Technique and Technologies*, **IV** (6), 91 (2015).